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Title: INCREMENTAL SAMPLING METHODOLOGY FOR SOILS AND SEDIMENTS [ASTM D 6323]

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1.0 Scope and Application

1.1 The purpose of this procedure is to obtain sub-samples from client provided samples which represent the concentration of the analytes of interest in the entire parent sample. This is based on the guidance in ASTM Standard D-6323 "Laboratory Subsampling of Media Related to Waste Management Activities," a DoD Quality Systems Manual (QSM) requirement.

1.2 This procedure applies to soils, sediments and other particulate matter. This method is highly dependent on client provided Data Quality Objectives. This procedure presents the laboratory's standard approach, but details at all stages of this procedure can vary from project to project. All project-specific variations must be documented and approved in writing. It is important that the analyst always check special project instructions in LIMS before proceeding.

1.3 TestAmerica has used incremental sampling methodology (ISM) for non-volatile organics (e.g., explosives residues by Method 8330), prior to acid digestion for metals analysis (e.g., Method 3050), and prior to analysis for volatile organics collected in multiple increments in the field and preserved in methanol (e.g., Method 5035). It can be used for a wide range of other analytical methods as well. However, this procedure is not applicable to soil samples to be analyzed for volatile organic compounds in which the entire sample provided by the lab's client is used for a single analysis (see the lab's volatile organics SOP for those details).

1.4 This SOP addresses the pre-preparation of samples. The details of the twelve QC Elements, not otherwise addressed, are described in the associated preparation and/or analytical SOPs.

2.0 Summary of Method

2.1 For non-volatile analytes, the entire sample received from the client is air dried to a constant weight. Large non-representative pieces (rocks and twigs that will not pass through the sieve) may be removed manually. Other extraneous materials are removed by sieving. A mortar and pestle or sieve shaker or mechanical grinder may be used to break up soil agglomerates during the sieving process. Depending on the analytical method to be used after subsampling and project objectives, the sample may be ground. The grinding options available at the laboratory include the ring-and-puck mill. A subsample is then taken using a multi-incremental approach.

2.2 ISM for Metals Analysis - the routine approach is to air dry, sieve to sub-10 mesh (2.1 mm), and collect 10 gram subsamples using 30 increments. The expectation is that the variability due to subsampling error will then be no more than 15% relative standard deviation (RSD) (see ASTM D-6323 for explanation and guidance for other acceptable variations). The Method 3050B digestion reagents are then increased proportionally to maintain the same chemistry as is used for 1 gram subsamples.

2.3 ISM for Explosives Analysis - the routine approach is to air dry, sieve to sub-10 mesh, grind, and collect 10 gram subsamples using 30 increments. If the samples are from firing points, then ring-and-puck grinding is required. The goal is to achieve 10% or less

RSD from subsampling variability. Further details for explosives are given in SOP DV-OP-0018 and are not discussed in this SOP.

2.4 ISM for Volatile Analysis – the multi-incremental sampling is done in the field, with the 30 increments of 5 grams are added to a septum-cap bottle containing 200 mL methanol provided by the laboratory. From that point on, the lab's procedure for medium level soils (SOP DV-MS-0002) is followed.

2.5 The basic formula to use when working with clients to select the optimal approach for other methods or other precision objectives is given in Attachment 1 to this SOP. The Attachment defines the trade off between subsample size, particle size, and the desired level of precision.

3.0 Definitions

3.1 Refer to the Glossary of the TestAmerica Denver Quality Assurance Manual (QAM) and policy DV-QA-003P, Quality Control Program, for definitions of general analytical and QA/QC terms.

3.2 Sample or Client Sample – refers to the entire quantity of material delivered to the laboratory for testing.

3.3 Subsample – refers to the portion of sample taken in the laboratory for a given analysis. The objective of this procedure is to ensure that the subsample is a reasonably accurate representation of the entire sample.

4.0 Interferences

4.1 If multi-incremental or equivalent systematic sampling processes are not employed in the field, then the extra laboratory effort entailed in this SOP may add little or no improvement in results.

4.2 Potential loss of lighter semi-volatile compounds (e.g., naphthalene) through the drying and grinding process has not been well studied. Before employing the procedure for such compounds, the possible loss of lighter compounds should be discussed with the client and if possible, investigated before the procedure is performed.

4.3 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or contamination causing misinterpretation of results. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by running blanks.

4.4 Contamination by carryover can occur when a low concentration sample is processed immediately following a high concentration sample. For this reason, special care must be taken to follow the equipment cleaning steps.

4.5 As described in this SOP, the lab does not routinely grind samples for metals testing. It is expected that detection limits and reporting limits for some metals would have to be elevated based on long-term blank results if grinding is required.

5.0 Safety

5.1 Employees must abide by the policies and procedures in the Environmental Health and Safety Manual, TestAmerica Denver Addendum to the Environmental Health and Safety Manual, Radiation Safety Manual and this document.

5.2 This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, nitrile or latex gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

5.3 Specific Safety Concerns or Requirements

5.3.1 Anyone working in the grinding room needs to be enrolled in the Hearing Conservation Program. See DV-HS-0010 for details. Personnel operating grinding equipment are required to wear ear plugs when the equipment is turned on. When standing next to the Humbolt mechanical grinder described in Section 6.1.7 during operation, the decibel levels are above 80 decibels, therefore anyone operating the grinder must be enrolled in the Hearing Conservation Program and wear hearing protection. While the grinder is running, the decibel levels in the room are below 80 decibels, therefore personnel not enrolled in the Hearing Conservation Program can be in the room. Hearing protection is always available to every analyst and they are encouraged to use it.

5.3.2 Operations involving the handling of samples outside of sealed containers, e.g., sieving, are conducted in ventilation hoods to avoid exposure to dust. Dust masks are available for use in the grinding room, but are optional.

5.3.3 Operations involving the grinding of radioactive samples can be particularly hazardous due to the increased potential for exposure from airborne dust. If a sample is labeled as “CAT 1”, “CAT 2”, “CAT 3” or “CAT 4” and requires grinding through the ring and puck, contact the RSO immediately.

5.4 Primary Chemical and Material Hazards – cleaning solvents

MATERIAL	HAZARDS	EXPOSURE LIMIT (1)	SIGNS AND SYMPTOMS OF EXPOSURE
Acetonitrile	Flammable Poison	40 ppm – TWA	Early symptoms may include nose and throat irritation, flushing of the face, and chest tightness. Prolonged exposure to high levels of vapors may cause formation of cyanide anions in the body.
Acetone	Flammable	1000 ppm – TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
(1) Exposure limit refers to the OSHA regulatory exposure limit.			

6.0 Equipment and Supplies

6.1 Equipment

6.1.1 Balance, capable of measuring ± 0.01 g. Calibration checked per SOP DV-QA-0014

6.1.2 Sieve Shaker – used to facilitate the sieving of large sample volumes.

6.1.3 Ring and Puck - The grinding bowl and puck are cleaned after each use by washing with soap and water with a plastic brush, rinsing with hot tap water, rinsing with DI water, and then rinsing with a 10% acetonitrile solution in acetone. A final wipe down of the bowl and puck while still wet with solvent is done with a Kimwipe.

6.1.4 Trays – "baker's rack" type stack for air drying soils

6.1.5 Sieves - 10 and 30 mesh, brass for general use, stainless steel for metals testing. Sieves are cleaned after each use by washing with soap and water with a green plastic brillo pad (be careful not to damage the mesh), rinsing with hot tap water, rinsing with DI water. Prior to use, the sieves are rinsed with 10% acetonitrile in acetone and wiped with a Kimwipe. Sieves are allowed to dry in a hood prior to use.

6.1.6 Mortar and pestle – Porcelain, various sizes cleaned after each use by washing with soap and water, rinsing with hot tap water, and then rinsing with DI water. Prior to use, the mortars and pestles are rinsed with 10% acetonitrile in acetone and wiped with a Kimwipe and allowed to dry in a hood prior to use.

6.1.7 Mechanical Grinder – Humbolt Manufacturing Part Number H-4199. Used in place of a mortar and pestle to quickly reduce cakes of dry soil. The stainless steel grinder reduces soil agglomerates and sieves the soil through a 10 mesh sieve. The mechanical grinder is used to break up soil agglomerates, but it is not an alternative to the Ring and Puck. The mechanical grinder is cleaned after each sample by removing the hopper. The hopper is washed with soap and water, rinsed with tap water, rinsed with DI water, and then rinsed with 10% acetonitrile in acetone. The Hopper is then wiped dry with a laboratory tissue. The hammers and body of the grinder are cleaned after each sample by rinsing with DI water and wiping dry with a laboratory tissue.

6.2 Expendable Supplies

6.2.1 Plastic sample scoops – square-ended

6.2.2 Aluminum foil and aluminum dishes

6.2.3 Parchment paper to line trays for metals testing

6.2.4 Alconox detergent

6.2.5 Ottawa Sand – blank media for organics

6.3 Computer Software and Hardware

Please refer to the master list of documents, software and hardware located on R:\QA\Read\Master List of Documents\Master List of Documents, Software and Hardware.xls or current revision for the current software and hardware to be used for data processing.

7.0 Reagents and Standards

7.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

7.2 10% Acetonitrile in Acetone – mix 100 mL of acetonitrile with 900 mL of acetone. This solution is used for rinsing purposes only so exact measurements are not required.

8.0 Sample Collection, Preservation, Shipment and Storage

Container Type	Preservative	Holding Time
Plastic or glass	By individual test *	By individual test *

After air drying, samples can be stored at room temperature. As a secondary option, after air drying, the samples can be placed directly into ziplock bags and stored in the refrigerated cooler.

* - Reference the analytical SOPs.

9.0 Quality Control

9.1 The minimum quality controls (QC), acceptance criteria, and corrective actions are described in this section. When processing samples in the laboratory, use the LIMS Method Comments to determine specific QC requirements that apply. For SOPs that address only preparation, QC acceptance limits on the analytical results are not included. Refer to the appropriate SOP that describes the determinative method.

9.1.1 The laboratory's standard QC requirements, the process of establishing control limits, and the use of control charts are described more completely in TestAmerica Denver policy DV-QA-003P, *Quality Control Program*.

9.1.2 Specific QC requirements for Federal programs, e.g., Department of Defense (DoD), Department of Energy (DOE), etc., are described in TestAmerica Denver policy DV-QA-024P, *QA/QC Requirements for Federal Programs*. This procedure meets all criteria for DoD QSM 5.0 unless otherwise stated. Any deviation or exceptions from QSM 5.0 requirements must have prior approval in the project requirements.

9.1.3 Project-specific requirements can override the requirements presented in this

section when there is a written agreement between the laboratory and the client, and the source of those requirements should be described in the project documents. Project-specific requirements are communicated to the analyst via Method Comments in the LIMS and the Quality Assurance Summaries (QAS) in the public folders.

9.1.4 Any QC result that fails to meet control criteria must be documented in a Nonconformance Memo (NCM). The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. This is in addition to the corrective actions described in the following sections.

9.2 Grinding Blanks

9.2.1 Ring and Puck Grinding Blanks.

Before each sample is processed through the ring and puck mill, the ring and puck will be cleaned per Section 6.1. Then approximately 200 g of Ottawa sand will be ground. This ground sand will be saved and labeled with the sample ID of the next sample ground with the suffix “blank”. After a batch of samples has been processed through the ring and puck, a composite will be generated using sub-aliquots from all blanks ground before the samples. This is done by placing approximately 1 tablespoon of material from each of the individual sample blanks in a clean re-sealable plastic bag. The bag is then sealed and the material is mixed and homogenized by shaking and kneading the bag. A 10 g aliquot is then removed from the bag and labeled as the batch grinding blank. This composite is extracted and analyzed in the same manner as the field samples.

Corrective Action: If the composite grinding blank results are greater than the acceptance limits, then the individual grinding blanks will be extracted and analyzed to determine when the contamination occurred and exactly which samples were affected. Samples associated with a contaminated grinding blank with positive results for the same contaminant must be reprocessed and reanalyzed. If un-ground sample is not available, then the potential carry-over between samples must be described in a non-conformance memo and discussed in the final report case narrative

9.3 Precision

9.3.1 On a project basis, the lab will discuss precision objectives with the client prior to initiating work. If evaluation of the RSD is needed, the laboratory will need to analyze at least one set of triplicate samples in every preparation batch. In other cases, the lab will employ duplicate matrix spikes and control limits will be expressed as relative percent difference (RPD).

9.3.2 If the client supplies multiple field samples to use for replicate testing, then the laboratory will compare results to acceptance limits and qualify data if the precision limits are not met. If the replicates are prepared from the single field sample that is dried, ground, and sieved, then the acceptability of each grinding batch can be controlled based on the precision objectives established for the project.

9.4 Other QC samples (method blank, LCS, and MS/MSD) are created after subsampling, and vary depending on the analytical method. See DV-OP-0018 for special QC requirements for explosives, which usually require grinding a standard reference material.

10.0 Procedure

10.1 One-time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using an NCM. The NCM is automatically sent to the laboratory Project Manager by e-mail so that the client can be notified as appropriate. The QA group periodically reviews NCMs for potential trends. The NCM process is described in more detail in SOP DV-QA-0031. The NCM shall be filed in the project file and addressed in the case narrative.

10.2 Any deviations from this procedure identified after the work has been completed must be documented in an NCM, with a cause and corrective action described.

10.3 Dry the Samples

10.3.1 The entire contents of the sample container must be processed. It is not acceptable to remove any aliquots until after the sample has been dried, sieved, and ISM performed. If the client requests aliquots to be taken before the sample is dried, sieved, and ISM performed, an NCM should be written to document this was done per client request.

10.3.2 Depending on the sample size, the samples are laid out in aluminum pans, or on large trays lined with aluminum foil to dry. Some clients may request metals analysis on the dried samples. In those cases, samples are laid out on parchment paper.

10.3.3 Spread the samples out in a thin layer to facilitate drying. Use a disposable wooden spatula to break up any clumps and agglomerates.

10.3.4 The tray or pan that the sample is laid out into is labeled with the sample ID. A second analyst checks to make sure that the labels on the tray or pan match the labels on the client sample container to ensure samples are not accidently mixed up. This check is documented in TALS.

10.3.5 Place the samples in a hood or well ventilated area at room temperature. Document in TALS the date and time the samples were laid out to dry. If the samples are very wet, a fan can be used to help facilitate the drying process, but care should be taken so that the air flow is not strong enough to cause cross-contamination between samples. An electronic temperature recording device records the temperature of the room and the data is downloaded weekly.

10.3.6 When the samples appear to be dry enough that they can be sieved without caking, subsample approximately 15 grams into an appropriate weighing vessel and record the exact weight, the date, and the time (see Attachment 3). Set this 15 gram aliquot (still in the weighing vessel) next to the rest of the drying sample. Take care to use an appropriate weighing

vessel for the analytical methods requested, as the aliquot removed in this step will still be included in the volume used for ISM (i.e. Do not use an aluminum weigh boat for samples requiring metals analysis).

10.3.7 After 2 hours, reweigh the aliquot in the same weighing vessel and record the exact weight, the date, and the time. If the weight of the sample is within 10% of the previous weight, proceed to Section 10.2.

NOTE: The procedure for verifying that samples have been dried to a constant weight, as described in the previous two sections, is included for DOD compliance. For non-DOD samples only, it is acceptable for an experienced analyst to verify by sight that the samples are dry in the interest of meeting turnaround times or holding times. In the case that the constant weight determination is not performed, an NCM must be generated for deviation from this SOP. However, the constant weight verification procedure must be performed and documented for all DOD samples.

10.4 Sieve the Samples

10.4.1 Clean the sieves prior to use following the instructions in Section 6.

10.4.2 Some samples may require the use of a mortar and pestle or a mechanical grinder to break up dried clumps. Refer to Section 6.1 on how to clean and rinse the mortar and pestles and the mechanical grinder before use.

10.4.3 Sieve the entire dried sample through the appropriate sized sieve. Care must be exercised not to eliminate soil agglomerates during this step. The soil can be broken into small pieces with a gloved hand or another instrument (a wooden spatula for example). If a gloved hand is used, care should be taken to change out gloves in between samples so not to cross- contaminate samples.

10.4.4 Remove large rocks, vegetation, and twigs that do not pass through the sieve. Mosses and other types of fine vegetation should be physically shredded while sieving to release trapped soil and residues. The only materials that should be eliminated by sieving are rocks and vegetation. All soil must be broken up to pass through the sieve.

10.4.5 Place any soil that does not pass through the sieve into a clean mortar. Break up soil agglomerates using the pestle. Or as an alternative use the mechanical grinder. Be sure to break up all soil so that it can pass through the sieve. Only extraneous material such as rocks and vegetation should be removed with the sieve. Describe all extraneous material that did not pass through the sieve in an NCM.

NOTE: Some clients may request the portion of the sample that did not pass through the sieve to be saved and weighed. Check Method Comments before discarding any sample material.

10.4.6 Collect all of the material that passes through the sieve on a clean piece of foil or parchment paper.

10.4.7 An automatic sieve shaker can be used to help facilitate the sieving of samples. A receiver pan is placed under a sieve and the sample is added to the sieve with 1 or 2 small grinding stones. Then a lid or another receiver pan for a second sample is placed on top. The stack is then clamped inside the sieve shaker for no more than 30 minutes. Inspect the samples to ensure that only extraneous material such as rocks and vegetation were removed with the sieve. If needed use a mortar and pestle to break up soil agglomerates. Describe all extraneous material that did not pass through the sieve in an NCM.

10.4.8 If metals analyses are requested on the sample, perform ISM on the portion of the sample that passed through the sieve at this time before proceeding to any grinding steps in Section 10.6. Refer to Method Comments, Sample comments, and Login Comments for instructions if any other tests besides metals analyses are to be performed on an un-ground aliquot before proceeding to Section 10.6

10.5 Incremental Sampling Methodology for Metals and other methods requested on un-ground material.

10.5.1 Remove the cap from a 100mL digestion cup and place on a balance and tare. The entire sieved sample is spread out on a sheet of parchment paper to a 1 cm thickness.

10.5.2 Using a disposable square-ended spatula, take an appropriately sized subsample by collecting at least 30 increments from random locations through the entire thickness, top to bottom, of the layer of ground material.

10.5.2.1 For methods 6010B, 6010C, 6020, and 6020A a 10 g-11 g aliquot is required for each sample and each MS/MSD sample. Collect one extra 10 g-11 g aliquot per sample in case re-digestion is needed.

10.5.2.2 For method 7471A and 7471B, a 3 g-3.3 g aliquot is required for each sample and each MS/MSD sample. Collect one extra 3g-3.3g aliquot per sample in case re-digestion is needed.

NOTE: Sub-out ISM samples will need to aliquoted into amber 40mL VOA vials and delivered to sample receiving with the appropriate paperwork. Aliquot size will vary and depends on the analysis needed.

10.5.3 Record the sample weight on the ISM Worksheet described in Attachment 2.

10.6 Grinding

The instructions in this section are to be used as a general procedure when grinding is requested prior to extraction and analysis for any method. Reference DV-OP-0018 for details on grinding samples for explosive samples.

10.6.1 Ring and Puck Mill Grinding –

10.6.1.1 See Section 6.1 on how to clean the ring and puck dish.

10.6.1.2 If the sample is logged for ring and puck grinding, a grinding blank per Section 9.3.1 consisting of baked Ottawa sand will be processed through the ring and puck dish before each sample. These individual blanks will be composited into one grinding blank for the associated samples and will be analyzed in addition to the normal extraction blank.

NOTE: When preparing the grinding blanks, it is not necessary to do five 60-second grinds. One 60-second grind of the Ottawa sand is sufficient.

10.6.1.3 After a grinding blank has been processed through a ring and puck dish, that blank is labeled as the blank associated to the next sample processed through that same dish. Do not clean the ring and puck dish after the blank.

10.6.1.4 In a hood, transfer the sample into a clean ring and puck dish. Do not overfill the dish (approximately 300 g of sample can fit in one dish). If needed, grind the sample in 300 g or smaller increments and recombine after all sample has been ground. The entire sample must be ground. Place the dish securely in the holder and close the door on the machine. Grind the sample in five 60-second periods with a one minute cooling time between grinds for a total of 5 minutes of grinding. Remove the dish and in a fume hood, open the lid and inspect the sample. It should be the consistency of flour. The consistency of the material is checked by pinching some between fingers of a gloved hand and feeling for grit and by looking for any un-ground fibers. If grit is detected or if fibers are observed, additional grinding is needed.

10.6.1.5 If the sample reaches a flour-like consistency before all 5 one-minute grinds have been completed, then it might be beneficial to not perform all 5 grinds in order to avoid excessive heat and to avoid packing the sample onto the side of the grinder. If the analyst inspects the sample and it has flour like consistency before all 5 grinds are completed, they can make the decision to stop after less than 5 grinds. A NCM should be written to document the deviation from the source method and the reasoning.

NOTE: During the one-minute cooling time, the dish should be placed in a shallow ice water bath to facilitate cooling. Be sure the bath is shallow enough so that water does not get inside the dish.

NOTE: If multiple 300 g increments are used for grinding and the sample is recombined, it has been shown in DU/TRL QC that the sample is non-homogenous. To re-homogenize the sample, place all volume in to a clean plastic bag, seal, and carefully shake the bag for 1-2 minutes until sample is homogenous. Lay out the sample back on the foil/parchement paper

10.7 Incremental Sampling after grinding.

10.7.1 Remove the cap from a 40 mL amber vial or other appropriate container and place on a balance and tare. The entire ground sample is spread out on a sheet of parchment paper or aluminum foil to a 1 cm thickness.

10.7.2 Using a disposable square-ended spatula, take an appropriately sized subsample by collecting at least 30 increments from random locations through the entire thickness, top to bottom, of the layer of ground material.

10.7.2.1 For explosives a 10 g to 11 g aliquot is required for each sample and each MS/MSD sample.

10.7.2.2 For other extractable methods a 30-33 g aliquot is common, but reference project instructions and method SOPs.

10.7.3 Record the sample weight on the ISM Worksheet.

10.8 Maintenance

10.8.1 Approximately once a month, the cover on the Ring and Puck should be removed and any dirt should be cleaned up.

10.8.2 When excessive wear is noted, replace the hammers in the Mechanical Grinder.

10.8.3 Occasional lubrication of the Ring and Puck clamp is needed.

10.8.4 The o-rings in the Ring and Puck dishes should be replaced when worn.

10.9 Troubleshooting

10.9.1 The Ring and Puck dishes are all slightly different depths. Therefore the clamp that holds them to the grinder does not fit snuggly on all dishes without the use of a pad. It is important to have a snug fit to ensure the dish lid seals tightly to avoid sample loss.

11.0 Calculations

Relative Standard Deviation

$$RSD = \frac{S}{\overline{X}}$$

Where: S = standard deviation

$\overline{X}$ = mean

12.0 Method Performance

12.1 Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL policy in DV-QA-005P. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method or program requirements require a greater frequency.

12.2 Limit of Quantitation Verification (LOQV)

The verification of the limit of quantitation (LOQ or LLOQ) is performed quarterly for work performed according to the DOD/DOE QSM 5.0 or for programs which require the use of Method 8270D, Revision 5. A blank matrix is spiked at 1-2 the laboratory RL and carried through the entire preparation and analytical procedures. Recoveries are assessed based on historical limits.

12.3 Demonstration of Capabilities

All personnel are required to perform an initial demonstration of proficiency (IDOC) on the instrument they will be using for analysis prior to testing samples. On-going proficiency must be demonstrated annually. IDOCs and on-going proficiency demonstrations are conducted as follows.

12.3.1 Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation. The concentration of the QC check sample should be equivalent to a mid- level calibration.

12.3.2 Calculate the average recovery and standard deviation of the recovery for each analyte of interest.

12.3.3 If any analyte does not meet the acceptance criteria, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. TNI 2009 requires consecutive passing results. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

12.3.4 Until the IDOC is approved by the QA Manager (or designee); the trainer and trainee must be identified in the batch record.

12.3.5 Further details concerning demonstrations of proficiency are described in SOP DV-QA-0024.

12.4 Training Requirements

The Group Leader is responsible for ensuring that this procedure is performed by an associate who has been properly trained in its use and has the required experience. A new analyst must be working under documented supervision prior to approval of the IDOC. Documentation that a new analyst is performing under supervision must be entered into the batch record (View Batch Information) until that analyst's IDOC has been approved by the QA Manager (or designee). See requirements for demonstration of analyst proficiency in SOP DV-QA-0024.

13.0 Pollution Control

The use of organic solvents to complete the equipment cleaning steps is minimized. Quantities are limited to residues on equipment that quickly evaporate in a hood.

14.0 Waste Management

14.1 All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Environmental Health and Safety Manual for "Waste Management and Pollution Prevention."

14.2 The following waste streams are produced when this method is carried out:

Solid Waste – Waste Stream S

Flammable Solvent Waste – Waste Stream C

NOTE: Radioactive and potentially radioactive waste must be segregated from non-radioactive waste as appropriate. Contact the Radioactive Waste Coordinator for proper management of radioactive or potentially radioactive waste generated by this procedure.

15.0 References / Cross-References

15.1 "Guidance for Obtaining Representative Laboratory Analytical Subsamples from Particulate Laboratory Samples," USEPA, November 2003.

15.2 "Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities" ASTM D 6323-98 (Reapproved 2003)

16.0 Method Modifications

Item	Method	Modification
N/A	6323	N/A

17.0 Attachments

Attachment 1: Consideration of Fundamental Error in Selecting MIS Options
Attachment 2: ISM Worksheet
Attachment 3: ISM Constant Weight Worksheet

18.0 Revision History

Revision 10, dated 3 October 2017

- Updated section 2.1 to clarify air drying to a constant weight.
- Updated Section 8.0 to reflect the secondary storage option for air dried samples.
- Updated 10.3.6 and 10.3.7 to demonstrate drying to a constant weight.
- Update Section 10.5.2 to reflect ISM Sub-out practices.

- Added attachment 3

Revision 9, dated 28 February 2017

- Removed all references of the Ball Mill from the body of the instructions.
- Updated Section 3.1 to reference DV-QA-003P and QAM for general terms
- Updated Section 9.1 to be consistent with other SOPs
- Added current Sections 10.1 and 10.2 – NCM reference and instructions
- Updated Section 12 to include current MDL, LOQV, DOC and Training information
- Updated solid waste stream from D to S in Section 14.2

Revision 8, dated 29 February 2016

- Added aluminum dishes to Section 6.2
- Added Section 7.1 and 7.2
- Added what corrective action is used for Ring and Puck Grinding blanks that have hits in Section 9.4.1, as this differs from the corrective action for Ball Mill.
- Changed the acceptable weight range in Section 10.3.2.2 from 3-3.05 g to 3-3.3 g to be consistent with the 10% provided for other methods.
- Added more description to Section 10.4.2.2 to instruct the analyst not to wash the Ring and Puck dish at this step.
- Removed Section 10.6.5 that provided instruction about maintenance of the centrifuge as this equipment is not used in this SOP.
- Removed all revision histories 2010 and earlier (available upon request)

Revision 7, dated 28 February 2015

- Annual Technical Review
- Reformatted the SOP
- Added detail to Section 5.3.1 about the Humbolt mechanical grinder.
- Added information to Section 5 about the hazards of grinding radioactive samples.
- Revised Section 9.4.1 to give more detail on how the Ring and Puck composite grinding blanks are created.
- Revised Section 10.6 to include maintenance on the centrifuge.
- Added Attachment 2: ISM Worksheet

Revision 6, dated 05 February 2014

- Annual Technical Review
- Edited Section 6.1, subsection “Ball Mill” to allow for un-baked sand to be used in the cleaning of the ball mill stones and to allow the use of 1 pint cans.
- Edited Section 6.1, subsection “Sieves” to state a brillo pad can be used on the sieves so long as the mesh is not damaged.
- Added a comment to Section 9 stating that this procedure meets DoD QSM 5.0 criteria unless otherwise stated.
- Removed Acceptance Criteria information to Section 9. This information can be found in the analytical SOPs.
- Added a NOTE in Sections 10.4.1.1 and 10.4.2.4 giving instructions on how to ensure the sample is homogenous after it has been split into separate grinding

containers and then later re-combined.

- Added Section 10.6 Maintenance and Section 10.7 Troubleshooting per DoD QSM 5.0.

Revision 5, dated 31 January 2013

- Annual Technical Review
- Added Section 1.4 to address the 12 QC Elements.
- Updated Section 2.3 and 10.4.1 to allow the use of Ball Mill grinding. The laboratory successfully completed a Method Validation for Ball Mill grinding and therefore is able to offer this to all clients.
- Section 6 was updated to include cleaning procedures for sieves, Ring and Puck dishes, Ball Mill stones, Mortar & Pestles, and Paint Cans.
- Section 2.1, Section 6, and Section 10.2.2 were updated to include the mechanical grinder that can be used in place of mortar and pestle for samples that do not require metal testing.
- Section 9.3.3 was updated to include acceptance criteria for Grinding Blanks for DoD samples.
- Section 10.3.1 was updated to instruct the analysts to aliquot the samples directly into 100mL digestion cups for metals analysis.
- Removed Attachment 2: How to Batch Samples in LIMS.

Revision 4.2, dated 31 January 2012

- Removed all references to Multi-Incremental Subsampling which is now trade-marked.
- Updated Section 6.1 to reflect the correct number of small and large grinding stones used in the Ball Mill grinding of samples.
- Updated Attachment 2.

Revision 4.1, dated 20 January 2011

- Added detail about the electronic temperature recording device that records the temperature of the room.
- Revised procedure to state that during the one-minute cooling time, the dish will be placed in a shallow ice water bath to facilitate cooling.
- Revised Attachment 2 to include the method Dry_Grind and more details on how to batch samples that are logged for both MULTI_INC and grinding methods.

Earlier revision histories have been archived and are available upon request.

Attachment 1

Consideration of Fundamental Error in Selecting MIS Options

The following formula given in ASTM D-6323 was used to produce the table that follows.

$$S^2 = 18 * f * e * d^3 / M_s$$

where,

S^2 = the relative variance of the contaminant concentration due to the fundamental error

f = shape factor, a dimensionless number, a value of 0.5 can be taken as typical (Pierre Gy, 1982)

e = the population's average density (g/cm^3). For this table a typical soil density of 2.5 g/cm^3 was used.

d = the diameter of the largest particle in centimeters, and

M_s = the mass of the sample in grams

Sample Mass and Maximum Particle Size to Achieve a Desired RSD

Subsample Mass (g)	Sieve Size (US Standard Mesh)	At 5% RSD Max Size (cm)	At 10% RSD Max Size (cm)	At 15% RSD Max Size (cm)
0.1	35	0.02	0.04	0.05
1	18	0.05	0.08	0.10
2	13	0.06	0.10	0.13
5	12	0.08	0.13	0.17
10	10	0.10	0.16	0.22
30	7	0.15	0.24	0.31
50	6	0.18	0.28	0.37
100	5	0.22	0.35	0.46

Attachment 2

ISM Worksheet

G:/QA/Edit/FORMS/Organic Prep Forms/MASTER ISM Spreadsheet_Rev1

ISM BATCH:

Use this spreadsheet to document alquot weights when aliquotting into digestion or extraction vessels. If aliquoting into a temporary vessel, no need to document the exact weight because the sample aliquot will be transferred and weighed at the time of analysis.

Login	Sample	Method -->											
			(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)	(g)
		ALIQUOT 1											
		ALIQUOT 2											
		ALIQUOT 1											
		ALIQUOT 2											
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Attachment 3

ISM Constant Weight Worksheet

Located: \\tafs\Lab2\Denver\Admin\QA\Edit\FORMS\Organic Prep Forms

DV-F-0070-10-03-2017-Rev 0

ISM Batch Number:

Use this spreadsheet to document drying samples to a constant weight.

			Analyst Initial Date/time 1	Analyst Initial Date/time 2	Dry?		Analyst Initial Date/time 3	Dry?	
Login	Sample	Tare weight (weight of weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 1, 2	Proceed with ISM?	Gross weight (g) (weight of aliquot and weighing vessel)	Change in weights 2, 3	Proceed with ISM?
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